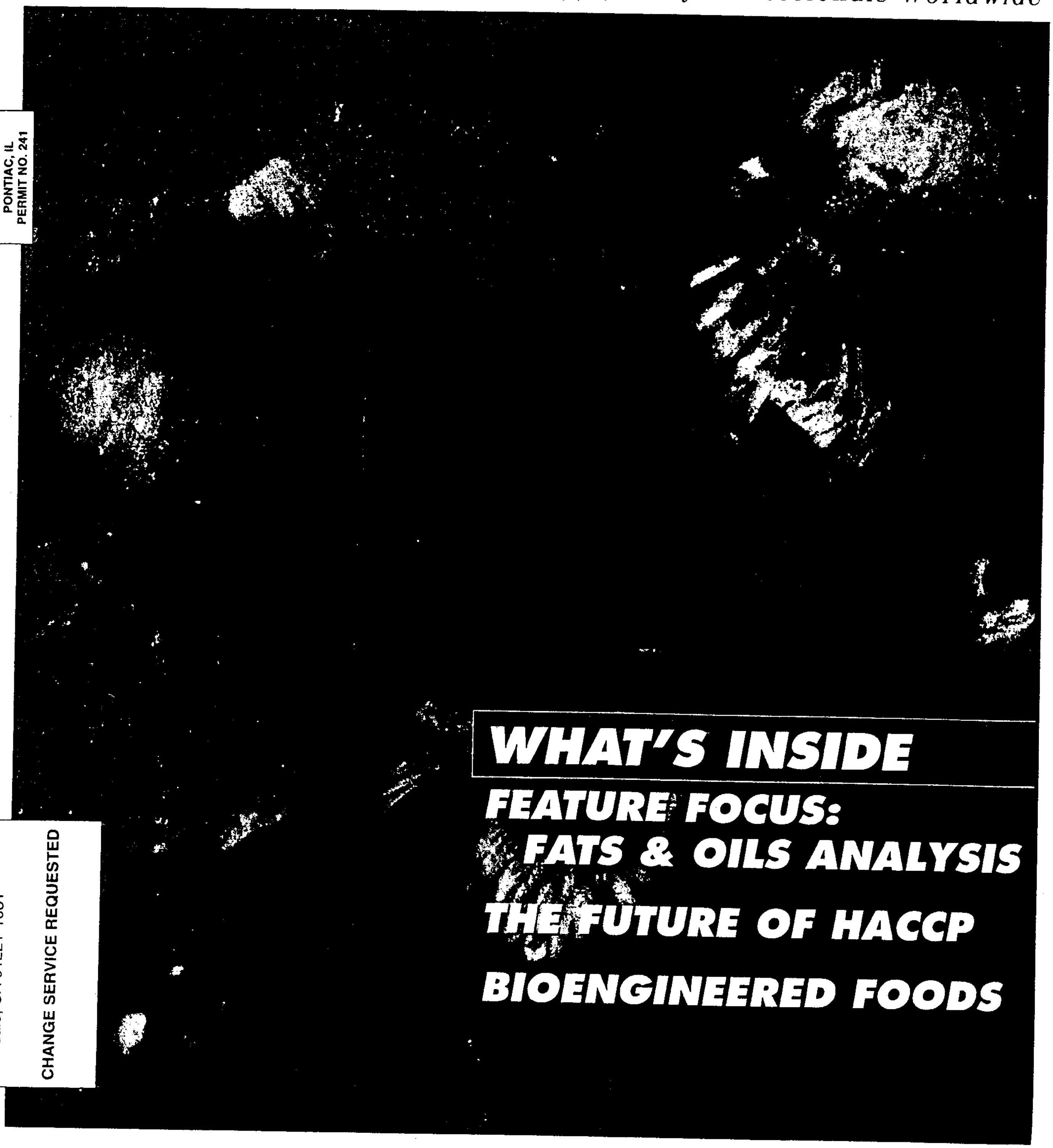
FOOD & TESTING ANALYSIS

February/March 1999
Volume 5, Number 1

Science-Based Solutions for Food Safety/Quality Professionals Worldwide



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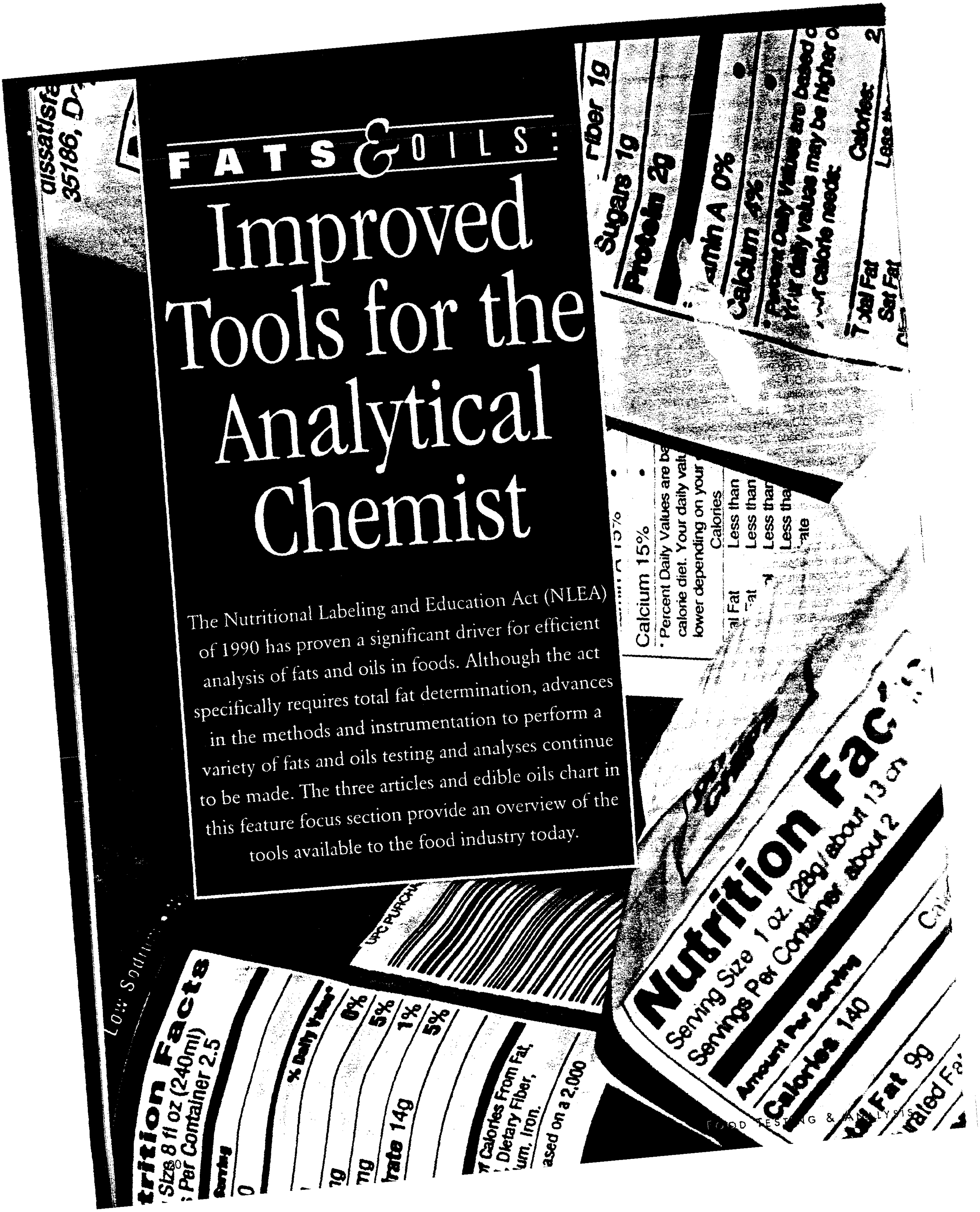

FATS & OILS:

Improved Tools for the Analytical Chemist

The Nutritional Labeling and Education Act (NLEA) of 1990 has proven a significant driver for efficient analysis of fats and oils in foods. Although the act specifically requires total fat determination, advances in the methods and instrumentation to perform a variety of fats and oils testing and analyses continue to be made. The three articles and edible oils chart in this feature focus section provide an overview of the tools available to the food industry today.

As part of this edition's feature coverage of fats and oils in FOOD TESTING & ANALYSIS *(FT&A) we are pleased to include the following interview conducted with two notable scientists involved in the research of fats and oils methods to provide some insight into some of the more recent advances made in this area.*

Since 1988, **Jerry W. King, Ph.D.**, has served as the lead scientist of the Supercritical Fluid Technology Group at the U.S. Department of Agriculture's (USDA) National Center for Agricultural Utilization Research Center (NCAUR). His research interests have included the development of supercritical fluid technology in food and agrimaterial processing, as well as for the analyses of toxicants, nutrients and oils/fats. He has authored more than 125 publications in supercritical fluid extraction (SFE), supercritical fluid chromatography (SFC) and related separation techniques. Among his numerous honors, King was awarded AOAC International's prestigious Harvey Wiley in 1997 for his research in analytical SFE.

Fred J. Eller, Ph.D., is a chemist in the Supercritical Fluid Technology Group at NCAUR, where his research interests include the analysis of fats in foods via SFE, particularly using GC-FAME analyses and the SFE of natural products. He has developed a number of fat analyses in conjunction with AOAC, AOCS and industrial companies. Eller is the author of more than 20 publications and two patents, including the isolation and identification of insect attractants.

FT&A: Since the determination of the amount of fat in foods is required for nutritional labeling information, the pressure to develop and improve analytical techniques is significant. What kinds of advances in analytical methods and tools have been made over the last few years, for what types of food products, and why?

King: There certainly have been a range of advances made in the area of fats and oils analysis in recent years. I would begin by saying that although there are new, heavily automated techniques available to the analytical chemist for fats and oils, there remains the classic considerations when dealing with a food matrix. In food matrices containing fats and oils, there is not necessarily one technique that will work for all analytes, even with advances in automated methods. I think we are narrowing the gap in terms of decreasing the proliferation of new or modified methods. However, the technique that is most useful still depends on the type of material being analyzed. For example, the oil and fat content of oilseed can be very easily analyzed using a particular technique, but the same technique applied to other products from which the oil is much harder to extract, such as pet foods, will not necessarily prove an easy analysis. The bottom line is that the analyst is still faced with applying basic food physical chemistry, and although new techniques are replacing classic liquid Soxhlet methods, there are still problems to be thought out.

One of the more recent advances centers on extraction methods. Supercritical fluid extraction (SFE) is a technique that was initially offered in the late 1980s as a method for use in the environmental industry. It wasn't until 1992 or 1993 that the food industry started appreciating the benefits of SFE due to pressures to reduce solvent usage in the laboratory. Analytical SFE is a technique that has helped to reduce solvent usage and improved methods, making analyses more rapid, and less expensive and labor-intensive. Also, some evidence exists that accceptance of technology is increasing, as shown by recent collaborative studies, for example, the published American Oil Chemists' Society (AOCS) method.

Other analytical developments during the last decade that have specific relevance to fat and oil analysis include: SFE analyzers; pressurized solvent extraction (PSE); microwave assisted extraction; and infrared (IR) and near infrared spectroscopy (NIR), including Fourier Transform infrared (FTIR). IR and NIR, in particular, are capable of determining a number of oil and lipid parameters, with and without prior extraction methods, and such IR-based techniques have made their mark in the lab. Also, for many years, nuclear magnetic resonance (NMR) analyzers have been used as rapid tools to measure parameters, such as the solid fat content of oils and fats. Of course, high-performance liquid chromatography (HPLC), gas chromatography (GC), and other methods are also used in the analysis of fats and oils.

FT&A: Ten years ago, there was a concentrated focus on analytical methods for omega fatty acids, followed by* trans *fatty acids. What do you think the next focus will be?

King: It's true that there's been a lot of effort put into developing analytical methods for omega fatty acids and *trans* fatty acids. With regard to the omega fatty acids, however, I'm not sure that we've achieved the optimal method for their determination. These can be particularly complex, for example, in fish oils where there are both omega-6 and omega-3 fatty acids that can make determination difficult. So these are additional areas in which methods research should be focused.

As for the future, conjugated linoleic acids (CLAs) appear to be an area in which additional analytical methods development will be a focus, due to their implication in human health and metabolism. The advent of increased interest in nutraceuticals, the latest food rage, as well as herbal and health foods, will likely require further fats and oils method development. Sterol esters, tocopherols and natural antioxidants are some of the components that will be of interest to analysts. For example, there is a Benecol® margarine substitute in the international marketplace (not in the U.S. yet) that contains cholesterol-lowering agents called sterol esters, hence the need for analytical methodology exists for these types of components. The vexing problem, though, is separating these from the triglycerides in a food matrix, which can be difficult because it can involve extensive sample clean-up or fractionation. I would say, that both the CLAs and the nutraceuticals will pose new challenges to the analyst in the fat and oil area.

FT&A: In your opinion, what are the most difficult food fats/oils to analyze? The least?

King: The most difficult are pet foods,

An interview with Jerry W. King, Ph.D. and Fred J. Eller, Ph.D.

high starch-containing matrices, and perhaps extremely specialized products containing phospholipids.

Eller: The least difficult are high-fat, low moisture products, such as snack foods like potato chips, or oilseeds. Low-fat or products with emulsifiers are difficult, as are products with high moisture content. Also, frequently extruded materials like pet foods can be difficult to extract fat from.

FT&A: What is your opinion on the importance of fatty methyl ester (FAME) analysis? How far have analytical techniques come in providing good data?

King: There's been a good 30 years in developing high-resolution gas chromatography columns for this field, so there's a lot of adequate GC separation technology available for precise FAME analysis. When this new method requirement was introduced in 1992, it was intended to replace less accurate gravimetric methods. The GC-FAME fat method proved to be a more sophisticated technology, but more labor-intensive. It is analyte-specific and requires acid hydrolysis, followed by an appropriate extraction technique formation of methyl esters, and then GC-FAME analysis.

Of course, as part of the Nutritional Labeling and Education Act of 1990 (NLEA), the GC-FAME method for determination of total fat for label compliance was required. In talking with a number of food companies, we found that, initially, many chose to have total fat analyses performed by contract labs. Also, a food label claim can be based on compositional analysis, but you should verify that analytically. Clearly, most food companies want to stay out of litigation and remain in regulatory compliance, so some have decided to develop standard methods in-house. Contract labs have developed some excellent NLEA fat methods, like those of Steven House and associates at Medallion Laboratories, in Minneapolis, MN, which are available to the food industry.

Eller: As Jerry says, determining total fat for NLEA compliance required a leap in method and technology. The U.S. Food and Drug Administration (FDA) modified the definition of total fat under NLEA, so you have to have knowledge of what you're analyzing before you can develop a method. GC-FAME analysis is by far the most accurate and specific means of determining fat in a given food. The GC analysis can also tell you how much saturated, mono-unsaturated and polyunsaturated fat is in a given matrix. This type of analysis can be used for all types of fats and oils, with minor adjustment of experimental conditions. It should be noted that while FAME analysis is very accurate, it is also not a trivial analysis to perform given the fact that the analyst must apply GC response factors for each fatty acid in the mixture to obtain the final result.

FT&A: Would you talk a little about the future of solvent extraction techniques as used in the analysis of fats and oils, with regard to solvent recovery issues being addressed by the U.S. government?

King: There are two government mandates developed in the past decade that have had a big impact. First, the U.S. Environmental Protection Agency's (EPA) Voluntary Toxics Release Inventory, which in theory mandated a 33% reduction in solvent use by 1993, and a 50% reduction by 1995. Included were many aromatic and chlorinated solvents.

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(continued from page 23)

Proficiency Program), which offers oilseeds and various oils and fats for selected food matrices, and pure oil standards.

Areas that need method development would include those that address sampling problems, particularly in the meat industry where sampling techniques are crucial. We've spent quite a bit of time analyzing ground beef, both gravimetrically and by the NLEA FAME method to show equivalency. Using SFE or the extraction technique, it taught us the importance of sample homogeneity.

Also, if you develop a standard reference material (SRM) based on a technique used 30 years ago, and then you develop a more accurate or improved technique that disagrees with the previously established value, it does not mean that the older value is necessarily the correct one.

Eller: One particularly good example of a SRM is NIST SRM 1544: Fatty Acids and Cholesterol in a Diet Composite, which we've used as a benchmark. Another is NIST 1546, a candidate reference material representing homogenous meat, which has potential to allow the determination of whether a fat analysis is being performed properly.

FT&A: Are there any trends toward harmonization or standardization of fats and oils methods to simplify instrumental oil analysis methods for contract labs or in-house food company labs to assure more representative results? For example, harmonizing current ASTM, AOCS and AOAC methodologies?

King: Yes. there is a tendency toward harmonization. What you're seeing is that an AOCS method will be accepted as an AOAC method; in other words, they have a tendency to interchange and recognize each other's methods. There is even increased involvement by the European Economic Community (EEC) toward standardized, collaborative methods, and I can foresee a harmonization on the international level.

If you look back a few years ago, the *Official Methods of Analysis of the AOAC International* listed 24 different methods for fat determination, depending on the food matrix. What has happened is that we've experienced close to 100 years of Soxhlet extraction, during which time people just made adjustments to the original method and propagated many methods over time. The NLEA GC-FAME could eliminate some of this proliferation of methods and extraction techniques. Analyzing the same foodstuff using the NLEA GC-FAME approach, you remove the ambiguity. And FAME's true merit is to try to come closer to the truth. Again, the real question is: What *is* the true fat content? Many companies are reluctant to depart from an established method to a new one, even if it's proven to be more accurate, since they want to match the traditional value.

Eller: As an example of that, I was speaking with a person a few months ago who was considering buying an SFE extractor for a food processing plant. The person asked, "Will it give me the 20.2% that I've always got by doing hexane extraction?" I said that it would be difficult to guarantee that result. So the person decided not to buy, because it might not give the same number they had always determined. The problem is that there's no guarantee that the older number is even correct in the first place. It's very important that methods and standard reference materials development for fats and oils continue in order to achieve a better standard of accuracy for food analysts and regulators.